

## Introduction

One of the improvements necessary to meet the Collider Run II performance goals is to improve the performance of the antiproton collection lens by increasing the magnetic field gradient from 750 Tesla per meter to 1000 Tesla per meter or higher.

A history of Fermilab solid lens service performance is provided below in Table 1. Improvements have been made in solid lens performance, primarily in external cooling water lines, the totality of which becomes apparent starting with lens 16. Early lens failures were due to external water leaks which were detected by cooling water system pressure and water level alarms. These early lenses could continue to be operated by periodic additions of cooling water to the cooling water systems and/or repairs to the cooling water lines. Eventually, the water leaks grew until continued operation was impractical. Starting with lens 16, the common failure mode in lenses with operating history shifts from external water leaks to internal water to lithium barrier failures (septum breach). The symptoms of the septum breach failures include high conductivity and high pH in the lens cooling water system which leads to an immediate end to the usefulness of the lens.

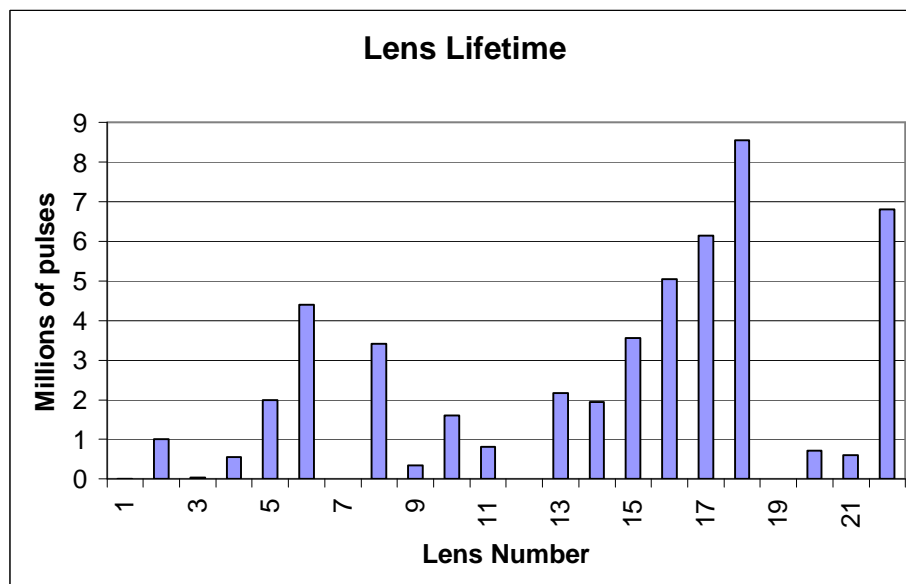


Figure 1

Many facets of lens construction and operation are being considered at this time with the goal of improving lens performance including lens assembly procedures, the lens fill procedure, lens test stand operation, choice of lithium isotope enrichment, helium and hydrogen gas production during operation, and more. It would be useful to disassemble failed operational lenses such as lenses 16, 17, 18, 20, and 21 to examine the septum breaches. An understanding of the location of the breaches along with metallurgical examinations of them may lead to an understanding of the cause of failures which may lead to development of an engineering solution. The purpose of this note is to describe a method of lithium removal so that examination of failed radioactive lenses may be pursued.

Removal of the lithium presents an opportunity to make a measurement of radioisotope production due to beam interaction with lithium. The quantities of radioisotopes produced in a lithium lens as a function of beam intensity was predicted [1]. A measurement has been made on the production of tritium and beryllium 7 on lithium in the target vault environment [2]. The results of the calculation and measurement may be used to predict the quantities of radioisotopes which may be present in lenses we propose to disassemble. Since all the lenses have been out of service for a minimum of 5 years, at least 33 half lives of beryllium 7 have passed so beryllium 7 may be neglected entirely. The anticipated quantities of tritium, neglecting radioactive decay by a half life of 12.3 years, as a function of failed operational lens is shown in Table 2.

Lens number	Removed from service	Total beam pulses	Average intensity per pulse	Total beam intensity (protons)	Tritium inventory (Ci) [ref 1]	Tritium inventory (Ci) [ref 2]
16	10/92	3,200,000	2E12	6.4E18	4	0.9
17	3/93	5,146,131	2E12	1E19	6.25	1.4

18	4/94	7,870,738	2E12	1.6E19	10	2.3
20	7/24/95	299,000	3E12	9E17	0.56	0.13
21	7/12/95	194,484	3E12	6E17	0.38	0.09

Table 2

Since lens 21 has been exposed to the lowest integrated intensity, it would be prudent to examine it first. In the course of the examination, tritium measurements would be made for comparison with the results of reference 1 and 2. It is very likely that any tritium produced in gaseous form in the lithium contour during beam operation escaped into the target vault through the water system when the lithium containment failed. The quantities of tritium which remain are thought to be due either to trapped interstitial gas or as a compound such as lithium hydride. The presence of the former is considered unlikely because of the diffusive nature of hydrogen gas. The compound LiH (MW 7.95,  $\rho$  0.82, mp 680°C), forms readily when Li is exposed to hydrogen at elevated temperatures. LiH reacts readily in water to produce hydrogen and LiOH. One may speculate that the effect of relatively intense ionization and elevated temperature which occurs during the beam and current pulse may encourage the formation of LiH. Like Li metal, LiH is also reactive in water and produces hydrogen gas [ref 3].

## Discussion

Lithium removal from a lens body will be accomplished in three phases.

In phase 1, the lithium lens body is placed in a glove box and an Argon atmosphere is established. Attachment 1 shows a cross section of a typical lens body. The total lithium volume contained by the lens body is approximately 100 cc. Complete removal of lithium by melting will not be possible because lithium wets the titanium and steel body surfaces. It is assumed that 90% of the lithium metal is to be removed by melting in phase 1. The lens body fill ports are

opened and the lens body is heated to 200 °C. Argon gas pressure is used to flow lithium out of the lens body into a steel dish where it is cooled. Tritium concentrations in the resulting Argon purge gas are monitored with a tritium gas detector (Triton or equivalent) during release. When lithium metal flow stops from the initial purge, the drain and purge operation is reversed to remove lithium metal from the low point at the opposite end of the lens body. Upon completion of lithium metal draining, lens body heating is discontinued while the argon gas purge continues to ensure a complete flow path through the lens body exists. Lithium removed from the lens body is weighed and an estimate of remaining lithium in the lens body is made. Lithium metal is placed in mineral oil in a suitable storage container.

In phase 2, it is assumed that 10% of the original lithium metal volume remains in the lens body. The argon atmosphere established in phase 1 continues without interruption. A tubing pump is connected to the lens body along with a hydrogen gas collection system. De-ionized, water (95% LCW) is used in the hydrogen gas collection system which basically consists of a Bell jar with appropriate plumbing. The phase 2 system is established in a way such that no oxygen is introduced into the collection system or glove box. Water is introduced into the lens body via the tubing pump. Hydrogen gas and LiOH solution are pumped/released into the Bell jar. The quantity of water introduced into the lens body is carefully controlled to limit the hydrogen production rate. The Bell jar is sized to accommodate at least twice the volume of hydrogen anticipated to be liberated in the water/lithium reaction.

The volume of water contained by the hydrogen gas collection system is estimated to be 20 times that required to react remaining lithium metal. The estimate is based upon the assumption that the Li reaction will occur with water until at least a saturated solution of LiOH is produced. When the lithium/water reaction appears to be complete, the lens body will be flushed with a quantity of fresh DI water. A soak period will be observed to allow additional lithium water reaction. Then the pH of the soak water will be checked. When the pH of the soak water is found to be below 9 to 10, the lithium metal can be considered to be completely consumed. The lens body can then be

drained. A combination of lens body heating and an Argon purge can be used to dry out the lens body. Finally, the lens body can be removed from the Argon atmosphere controlled glove box.

In phase 3, the hydrogen collection from the lithium water reaction in phase 2 is burned completely. The resulting water vapor is collected and is analyzed for tritium. The argon atmosphere in the glove box is replaced by air. A propane fueled Bunsen burner is used as flame source to burn hydrogen. The hydrogen is slowly and carefully introduced into the propane fueled flame. The resulting water vapor is drawn up into a heated copper chimney to prevent condensation. Heated water vapor is introduced into a water cooled Graham condenser where water from the combustion of propane and hydrogen is condensed. The water is collected at the exit of the condenser in an Erlenmeyer flask. The Erlenmeyer flask is of the filtration type and contains a side arm through which air is pumped to ensure positive flow of combustion gas and water vapor throughout the system. The air pump is a water powered filtration pump which develops suitable flow to transport water vapor through the copper chimney to the condenser.

In phase 4, system disassembly and handling of LiOH solution is addressed.

## References

1. Radiological Consequences of a Lithium Lens Rupture, pbar Note 454 , W. S. Freeman, 3/3/86
2. Lithium Irradiation Experiment, pbar Note 640, A. Leveling, 8/22/00
3. Rare Metals Handbook, Chapter 12, Lithium, P.E. Landolt

## Phase 1 Procedure

Refer to Attachment 2 for Phase 1 procedural steps.

NOTE: If a fire occurs during the unfilling process, evacuate the AP0 service building and call the fire department.

1. Attach individual purge and drain line arm assemblies to the lens body fill ports.
2. Apply insulation to the lens body surfaces to prevent excessive radiant heat loss into the glove box.
3. Mount lens body in insulated fixture with the beam axis aligned horizontally and with the fill ports positioned down.
4. Install lens body fixture, phase 1 components, and phase 2 components into the glove box.

NOTE: Purge all tubing with Argon gas prior to connecting to the tubing pump or gas collection system.

5. Establish an argon atmosphere in the glove box.
6. Connect the glove box vent to the gas inlet of the tritium gas sampler. The exhaust of the tritium gas sampler should be routed to an outdoor location.
7. Ensure tritium gas sampler is operational and connected to an appropriate data logging system (e.g., ACNET).
8. Open the lens body fill ports.
9. Open the argon purge port 1 and lens body drain port 1.
10. Apply argon gas pressure (nominally <10 psig) on purge port 1.

NOTE: Introduction of Argon purge gas to the lens body will cause argon gas to flow into the glove box.

11. Place a steel dish partially filled with mineral oil under fill port 1.

NOTE: The tare weight of mineral oil and steel dish should be collecting lithium.

12. Heat the lens body to approximately 200 °C.

13. Monitor tritium response throughout the Argon purge

14. Collect liquid lithium into 0.4 liter steel dish from drain port 1.

NOTE: Preheating the gas may be desirable to prevent lithium freezing in the lens body.

15. When lithium ceases to flow from the lens body, continue argon gas flow.

NOTE: The lens body and valves are insulated and may be handled safely with glovebox gloves.

16. Open drain port 2 and argon purge port 2.

17. Close argon purge port 1 and drain port 1.

18. Continue heating.

19. Collect liquid lithium in steel dish from drain port 2.

20. When lithium no longer flows from lens body, turn off lens body heaters.

21. Continue argon gas flow to aid in body cooling and to ensure flow path exists through the lens body.

NOTE: Argon gas flow may be arrested by lithium metal freezing in the purge pathway. If this occurs, energize lens body heaters and repeat steps 14 through 18 until argon gas flow is established in cooled lens body.

22. Allow lithium to solidify and cool in steel dish.

23. Remove steel dish containing lithium through the transfer compartment.
24. Note the mass Li metal collected in the steel dish and estimate the volume of lithium remaining in the lens body.
25. Transfer solid, cool Li metal to an appropriate covered, mineral oil-filled container for storage.
26. When lens body has cooled to room temperature, discontinue argon gas flow through the lens body.
27. Shut drain port #2 and purge port #2.
28. Allow Triton gas monitor to continue to operate until background counting rate returns.
29. Calculate the quantity of tritium released.

## **Phase 2 Procedure**

Refer to Attachment 3 for Phase 2 procedure setup.

NOTE: If a fire occurs during the unfilling process, evacuate the AP0 service building and call the fire department.

1. Maintain Argon atmosphere in glove box throughout Phase 2 procedure.
2. Connect tubing pump and hydrogen gas collection system to the lens body.

NOTE: The water system is vented into a Bell jar so that hydrogen gas pressure will not build up and blow water line connections from the circulating water system.

NOTE: The total water volume required to circulate through the lens body is based upon the amount of lithium remaining in the lens body. For example, 1 liter of water should be sufficient to react 10 cc of lithium in the lens body and provide sufficient cooling. Since the total circulating water volume is approximately 7



gallons, the resulting LiOH solution should not be saturated thereby preventing obstruction of the water path by precipitates of LiOH. Approximately 41 kcal heat is produced and assuming no heat transfer to the lens body would raise the temperature of 1 liter of water 41 degrees C.

Caution: Assuming 10 cc of lithium metal remain in the lens body, 8 to 9 liters of H<sub>2</sub> gas will be produced due to the water reaction with lithium metal. This gas is to be stored in the Bell jar for use in phase 3.

Caution: In steps 4 through 12, hydrogen gas will be produced and collected in the Bell Jar. If at any time, the production of gas becomes excessive, discontinue operation of the tubing pump and allow the hydrogen gas production to subside before proceeding.

NOTE: the water pH will increase from 7 to 14 as potentially radioactive lithium metal reacts with circulating water. Use appropriate precautions when handling caustic, potentially radioactive solutions.

3. Open lens body drain port #1 and #2.
4. Introduce approximately 10 milliliters of water into the lens body with tubing pump
5. Note release of hydrogen gas from lens body by formation of gas bubbles in Bell jar. Note time of introduction of water.
6. When evidence of gas production subsides in the Bell jar, note time.
7. Introduce 10 milliliters of water into the lens body with tubing pump.
8. Repeat steps 7 through 9 until 100 milliliters of water have been added.
9. Calculate the average of times recorded in step 8.

10. Establish a flow rate of 10 milliliter per average time found in step 9 through the pump body with the tubing pump for a time equal to 20 times the average time found in steps 5 through 8.
11. Continuously monitor discharge tube in Bell Jar to monitor gas production.
12. Establish a 50 milliliter per minute flow rate through the lens body for 1.5 hours or until evidence of hydrogen gas production subsides.
13. Stop tubing pump.
14. Close valves in pumping circuit.
15. Close valve between supply side of pump from Bell jar reservoir and open valve to provide a fresh source of de-ionized water.
16. Reroute tubing to include the septum cooling water channel in series with the lens body.
17. Open valves in pumping circuit.
18. Pump 2 liters of clean DI water through the lens body and septum cooling water circuit and allow to discharge into Bell jar reservoir or an alternate container if necessary.
19. Stop tubing pump. Monitor discharge tubing for evidence of hydrogen production for 5 minutes.
20. If there is evidence of hydrogen gas production reconnect tubing pump to Bell jar reservoir and proceed to step 14.
21. If there is no evidence of hydrogen gas production, proceed to step 23.
22. Collect a 50 milliliter sample of water by momentarily energizing tubing pump and collecting the effluent in a beaker.

23. Check pH of sample and record the value.
24. If the pH exceeds 9, reconnect tubing pump to lens body and Bell jar and proceed to step 14.
25. If pH of sample is less than 9, assume that all lithium has been reacted and proceed to step 27.
26. Disconnect pump suction from Bell jar and direct discharge to Bell jar.
27. Energize pump to push remaining water out of lens body with Argon.
28. Stop pump.
29. Close valves on Bell Jar vent line to maintain gas volume.
30. Disconnect discharge line from lens body to Bell Jar vent line.
31. Reverse pump direction and direct pump suction to Bell jar.
32. Energize pump to remove water from low point.
33. Stop pump
34. Establish an argon gas purge through the lens body and septum cooling water circuit to dry out the lens body assembly.
35. Close drain ports #1 and #2.
36. Remove lens body from glovebox.

### **Phase 3 Procedure**

Refer to Attachment 4 for phase 3 procedure.

NOTE: If a fire occurs during the unfilling process, evacuate the AP0 service building and call the fire department.

1. Ensure valves are closed on Bell jar tubing to preserve hydrogen gas collected in the Bell jar.
2. Establish an air atmosphere in the glove box.
3. Setup burner, chimney, and condenser on bench outside of glove box as shown in Attachment 4.
4. Establish cooling water flow to Graham condenser.
5. Establish air flow through chimney by energizing air pump or water filtration pump.
6. Light propane fueled Bunsen burner and establish a flame height of 2 to 3 inches by adjusting fuel inlet needle valve and air mixing sleeve.

CAUTION: the glass funnel and chimney will become very hot due to burner operation. Do not handle the glass funnel or chimney until after the burner gas flow has been stopped and these components have been permitted to cool.

7. Allow burner to operate for 5 minutes and note collection of water in Erlenmeyer flask.
8. Crack open Bell jar vent valve to slowly introduce hydrogen gas into Bunsen burner.
9. Continue introduction of hydrogen gas into Bunsen burner flame until all gas in the Bell jar is consumed and water just reaches check valve in the vent line.

NOTE: Add water to water tank if necessary to force hydrogen gas from Bell jar into burner flame.

10. Close Bell jar vent valve.

11. Stop gas flow to Bunsen burner and ensure flame is extinguished.
12. Allow chimney and funnel to cool. Collect water in Erlenmeyer flask for further analysis.

### **Phase 4 Procedure**

1. Pump neutralized LiOH solution from Bell jar reservoir to 5 gallon plastic carboys and dispose of in accordance with laboratory procedures.
2. Neutralize LiOH solution with HCl to pH range of 5 to 9.

NOTE: Assuming 10cc of lithium metal are removed by reacting with water, approximately 150 ml of 5 N HCl will be required.

3. Collect samples of neutralized solution for analysis of tritium and accelerator produced isotopes.

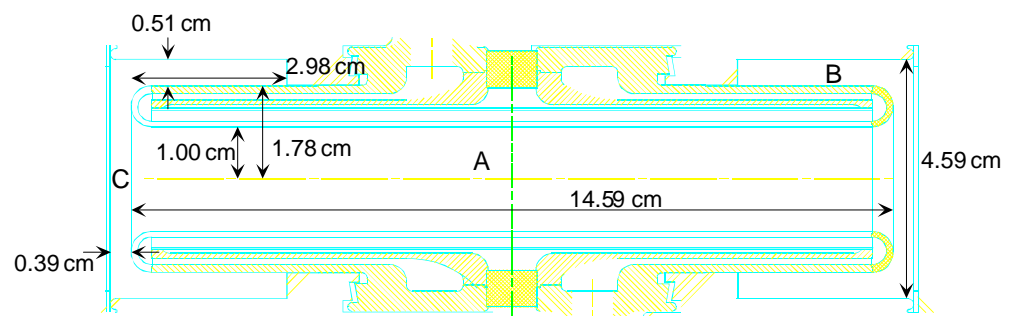
NOTE: Water used to dissolve lithium may need to be disposed of as radioactive waste depending on sample collected in Phase 2.

NOTE: Other lithium compounds may be present and fixed at the site of the breach. Removal of such materials should be made in a passive manner so that septum surfaces are not altered by this removal.

Pbar Note xxx

**Draft**

A Practical Method for Unfilling a Solid Lithium Lens



NOTE: Assume fill ports are negligible

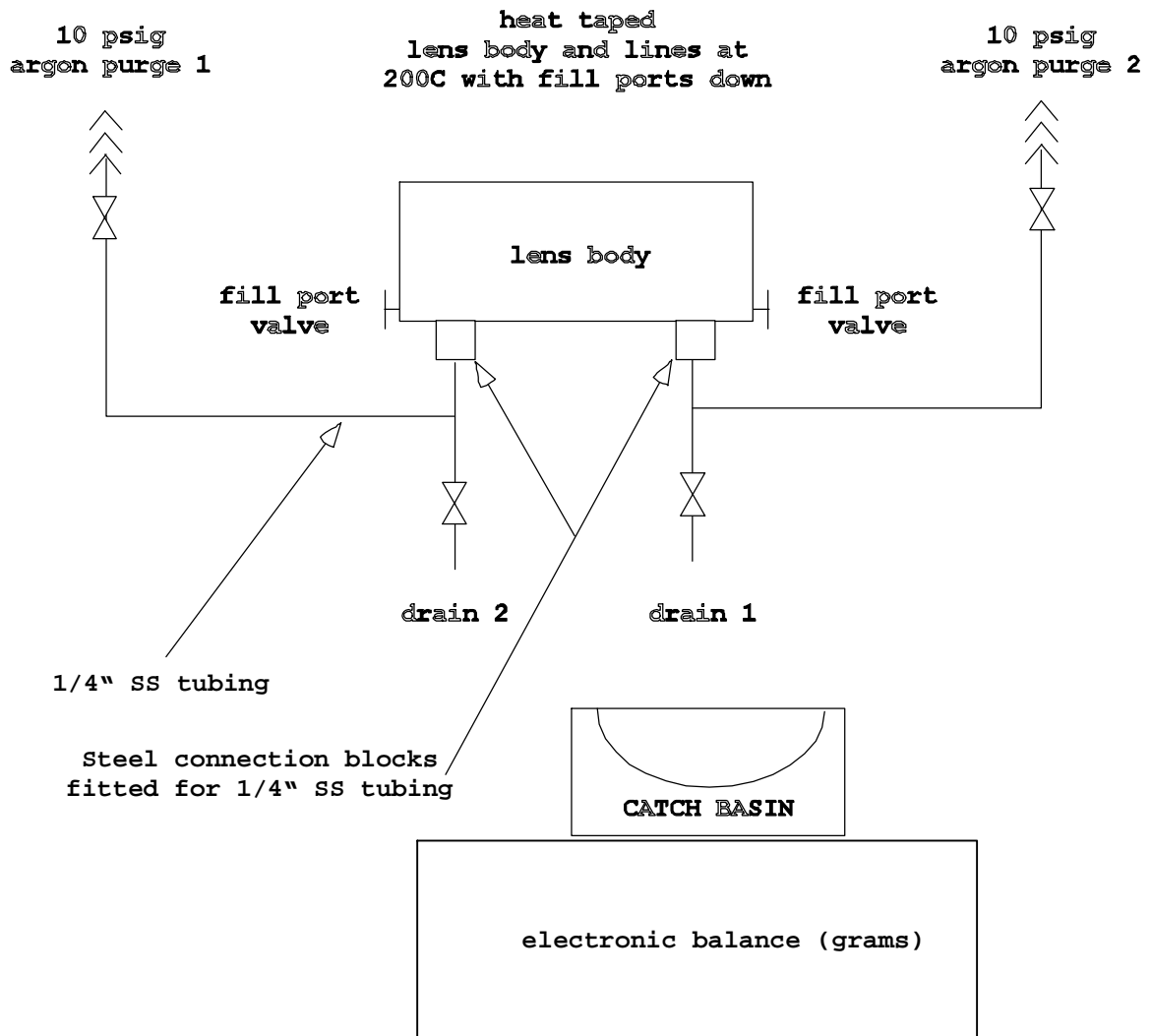
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Attachment 1

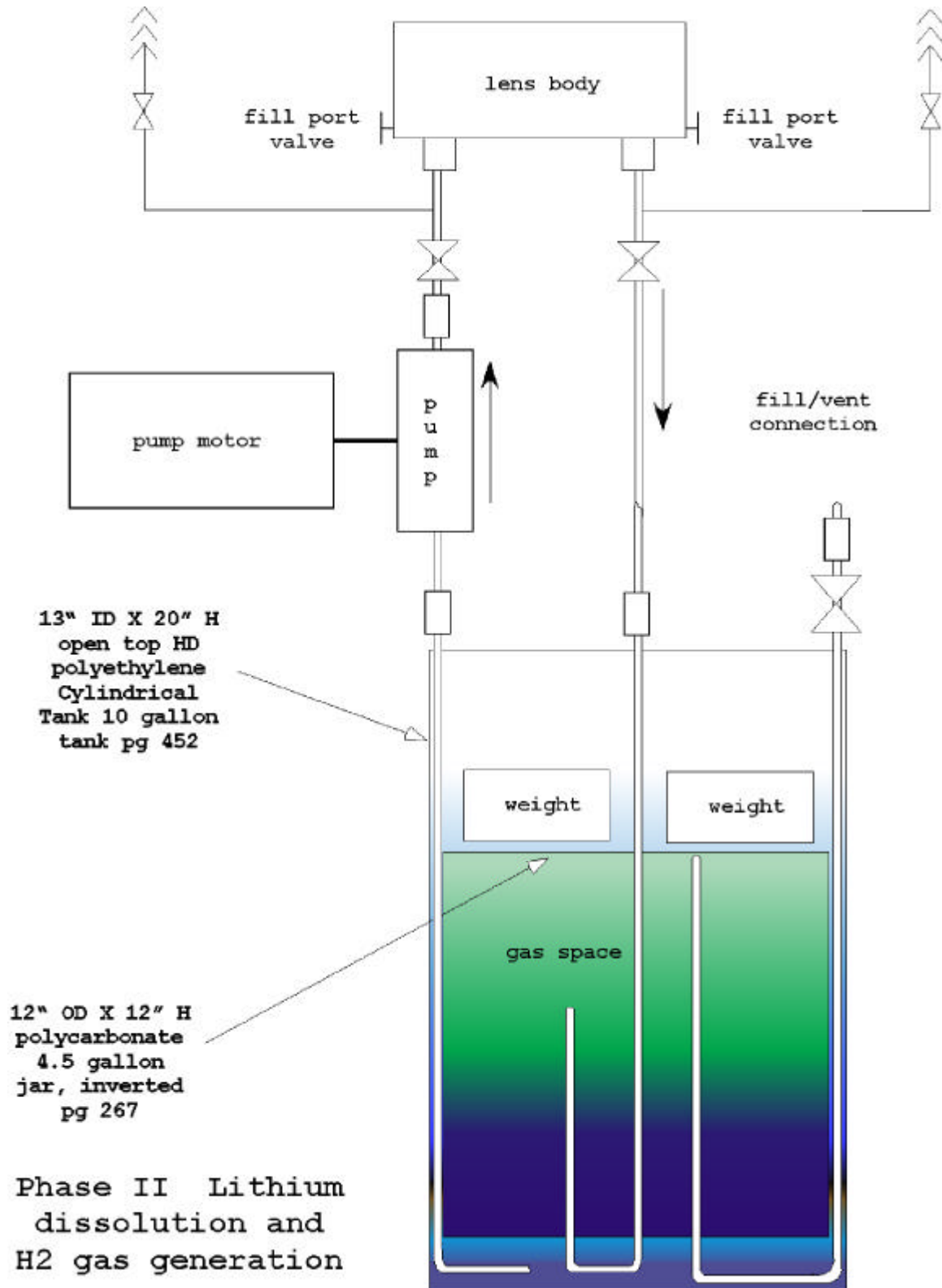
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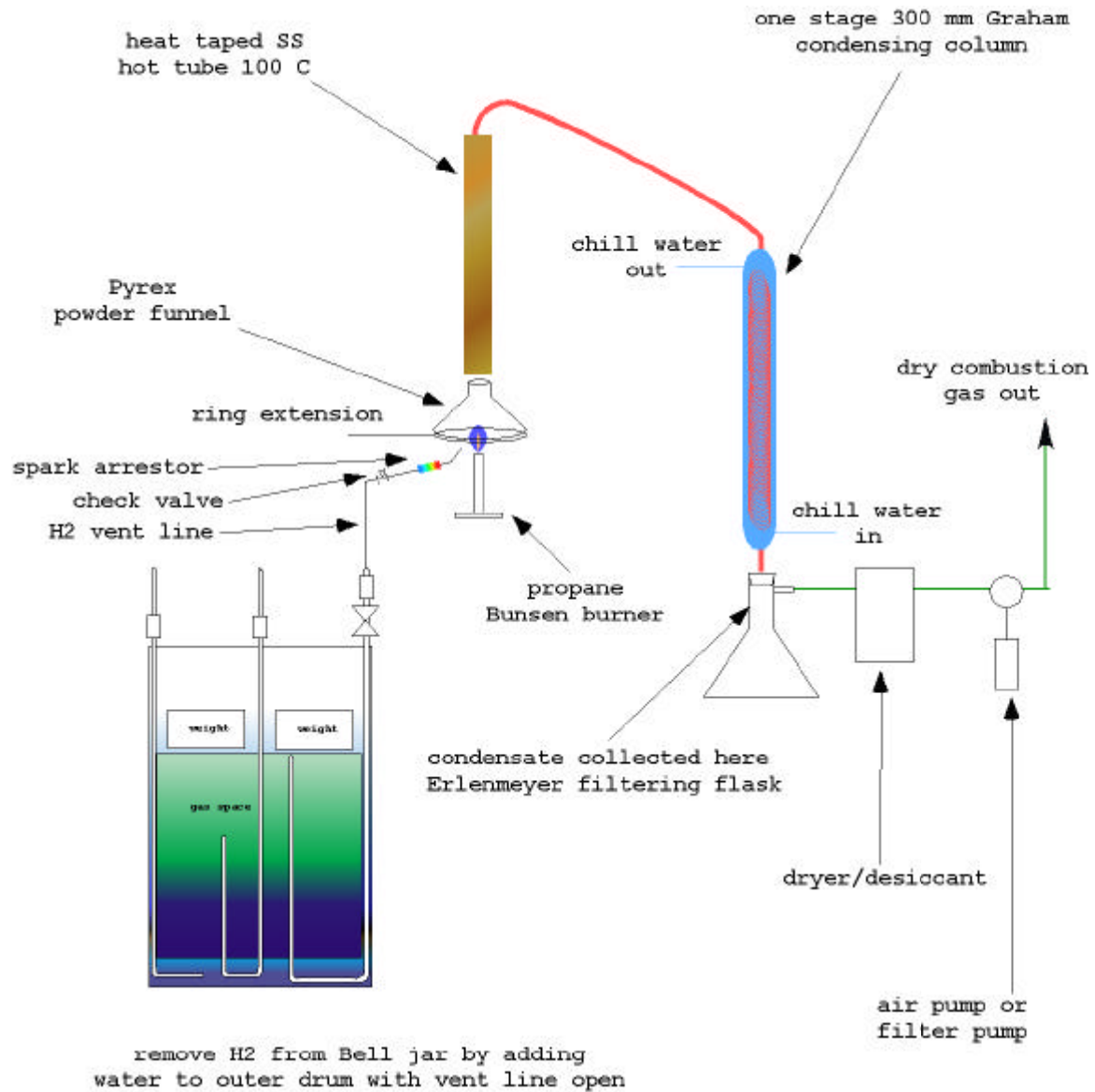
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**Phase I**  
**Lens Body Lithium Draining**







Collection Lens Body Unfilling Plan  
PHASE III